AIAA SciTech 2023

An Overview of Experiments in The Entry Systems Modeling Project

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Agenda

1) Varcum Oxidation Studies

- Microstructure of FiberForm
- Comparison of FiberForm Constituents

2) Arc-Jet / Material Response

- Spallation Testing at AHF

3) PICA-NuSil

- HyMETS Testing
- Preliminary Pyrolysis Gas Measurements

Differential Oxidation of FiberForm Constituents

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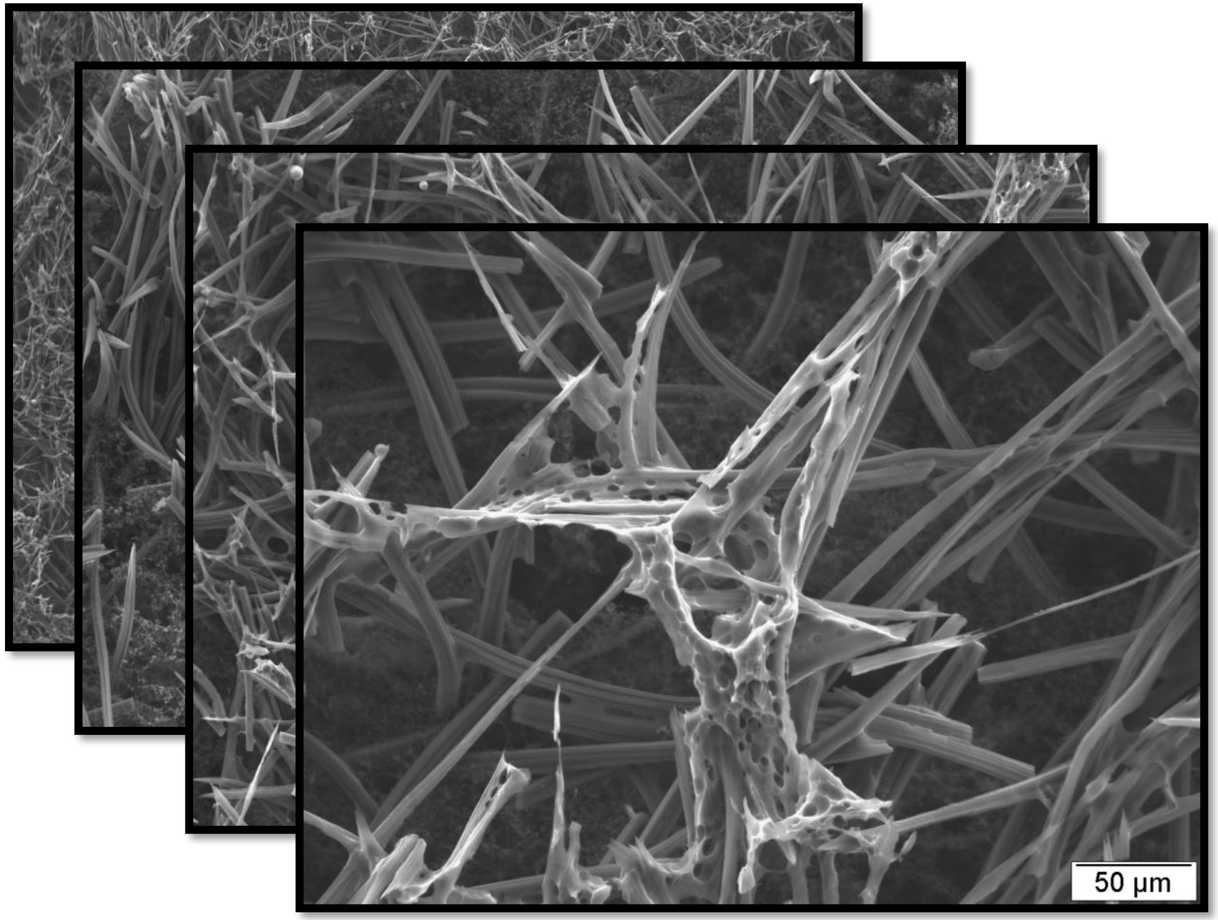
NASA POC: Brody Bessire

Differential Oxidation of FiberForm Constituents

S.E.M. Images of Charred PICA (PSO3)

(Presumed to be free of SiOC or SiO_2)



Credit: Jose F. Chavez-Garcia and Brody K. Bessire

- FiberForm is composed of a carbon fiber network bound together with a charred phenolic binder (varcum).
- PICA is manufactured by impregnating FiberForm with a phenolic resin (SC-1008).
- Charred PICA consists of regions of pyrolyzed phenolic resin and carbon fibers that have eroded due to oxidation.
- Question: Does varcum oxidize at a different rate than the carbon fibers of FiberForm?
- Question: If differential oxidation of FiberForm constituents is observed then is this a significant channel for spallation?
- First step: Isolate constituents of FiberForm and measure oxidation rates under well-controlled experimental conditions.

Isolating Constituents of FiberForm

- Carbonize varcum under relevant conditions and provide a surface that is amenable to spectroscopic measurements.

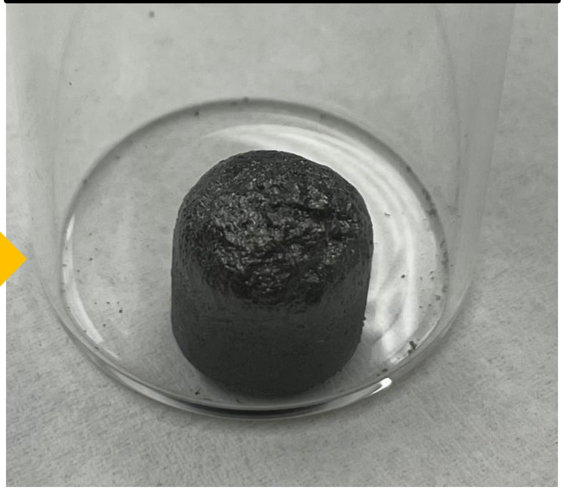
Cured Varcum

- **Cure Varcum Powder @ 160 °C for 30 minute.**
- **Crush cured varcum with mortar and pestle.**

High-Temperature Tube Furnace

- **Carbonize varcum powder in graphite crucible @ 1500 °C for 3 hours.**

Varcum Char Results



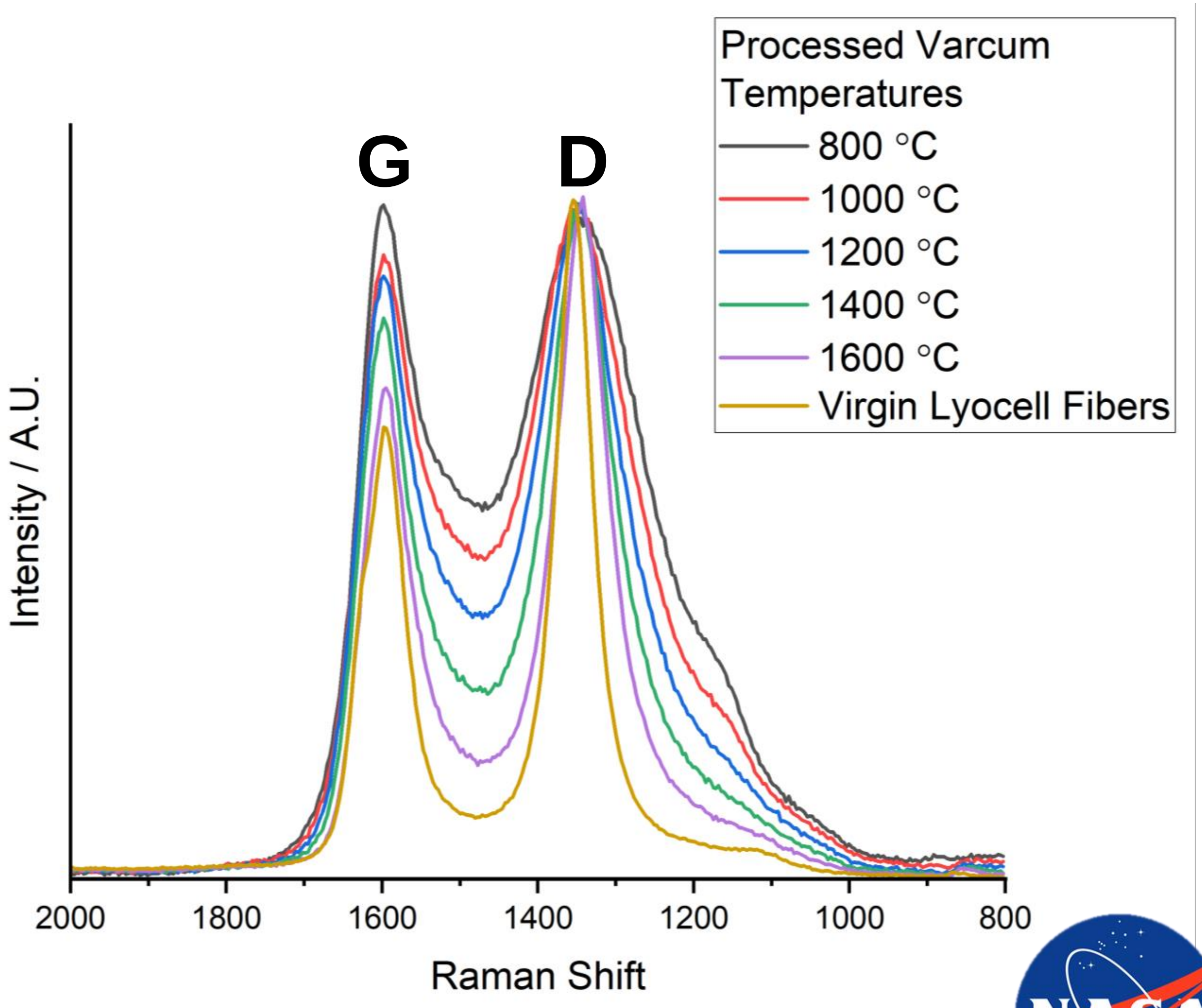
- **Process produces a pellet with a flat end.**

Raman Spectroscopy

- **“D” Peak at 1350 cm^{-1} increases in sharpness with increasing order.**

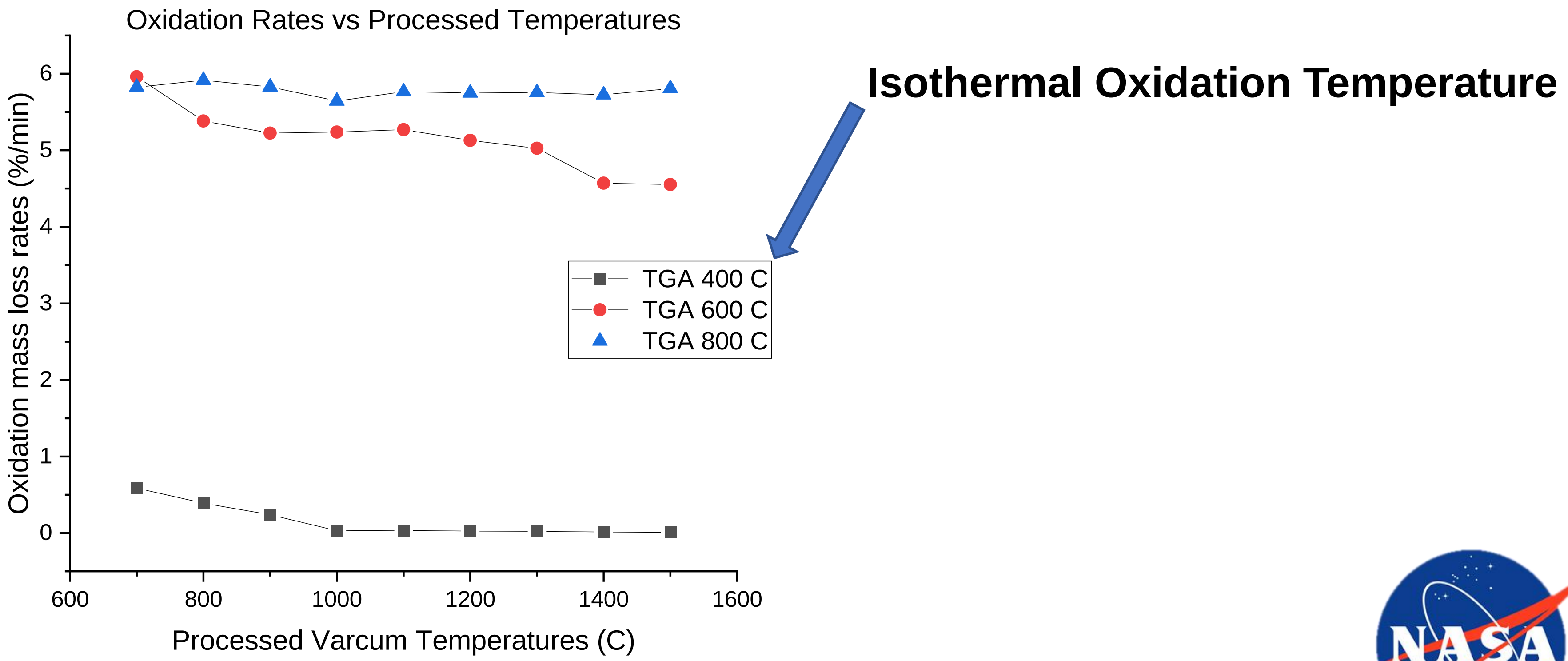
- **I(D)/I(G) ratio increases with heat treatment.**

- **Amorphous content decreases as peaks at 1200 cm^{-1} and 1350 cm^{-1} shrink.**



Thermogravimetric Analysis / Oxidation Rates

- **Oxidation rate generally increases with higher oxidation temperatures**
- **Peak oxidation rate increases and time to reach peak rate decreases with higher isothermal oxidation conditions**



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Spallation Experiments at AHF

Kristin Price, John R. Onan, Prof. Mike Renfro, Prof. Sean C. C. Bailey, & Prof. Alex Martin

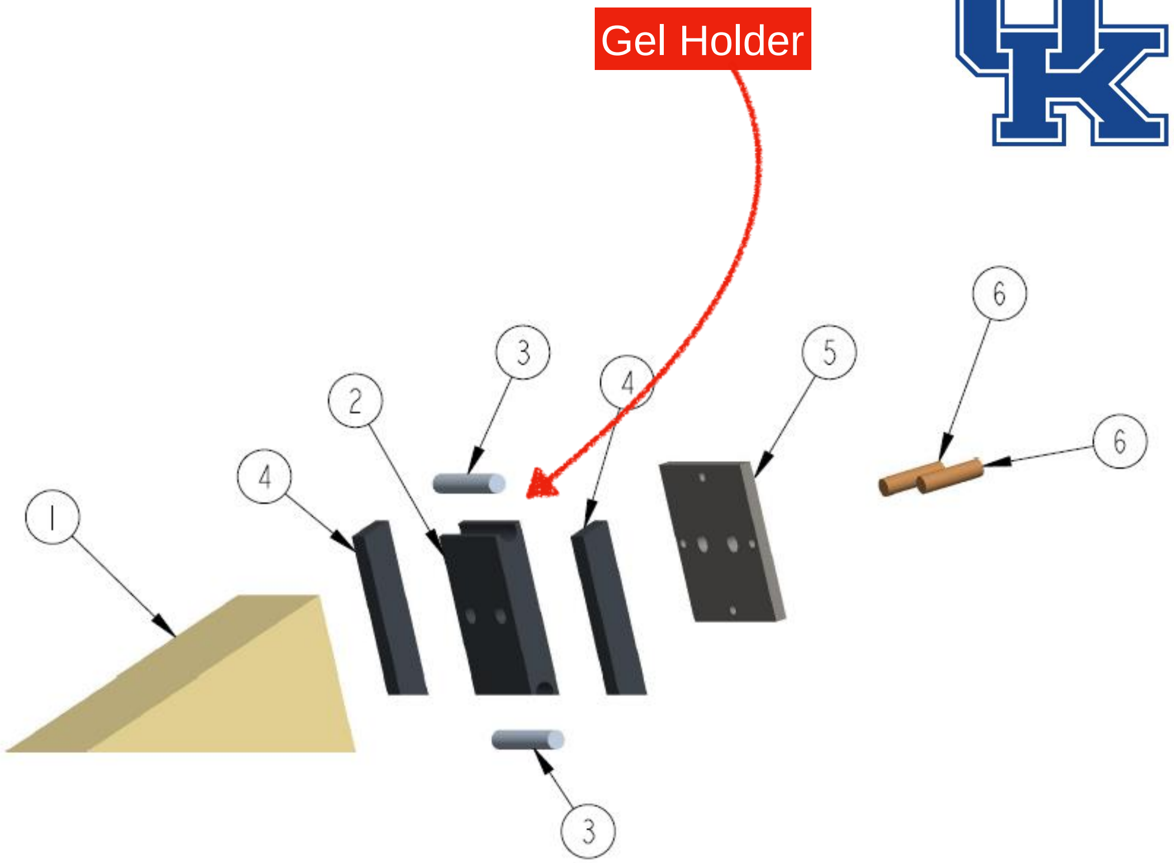
Mechanical and Aerospace Engineering, University of Kentucky

Kristen Price, "Characterization of spalled particles resulting from arc-jet tests" – tomorrow (TP-15)

NASA POC: Eric Stern & Brody Bessire

AHF Test Campaign / Spallation Test

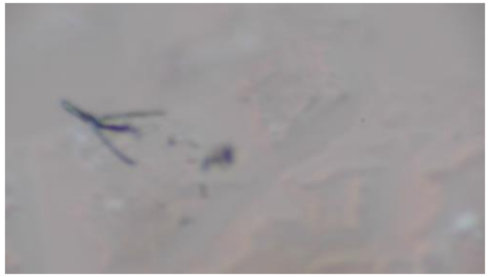
- **Aim**
  - **Capture spalled particles to identify size and weight**
- **Approach**
  - **16 PICA and FiberForm samples, at low heating air conditions**
  - **Particles will be collected by a reservoir at the back of the sample**
  - **Shadowgraph will be used to track particles**
  - **High-speed camera will be focused on ejection sites**

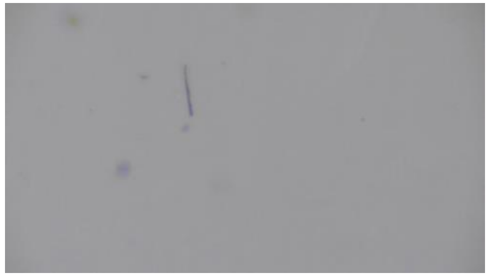


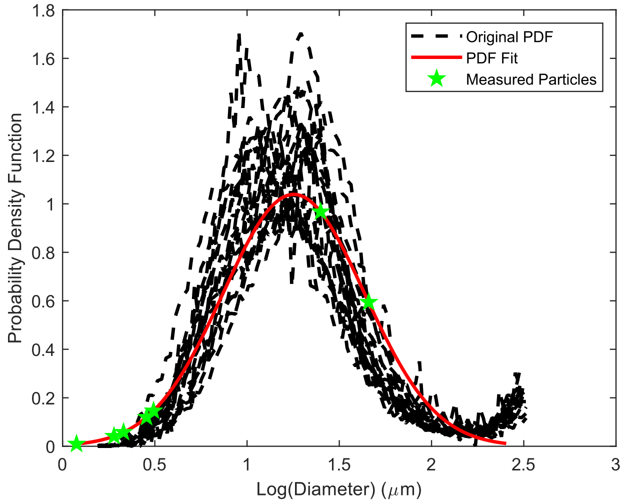
POC: Kristen Price, John, R. Onan, Sean C.C. Bailey, Alex Martin, Mike Renfro

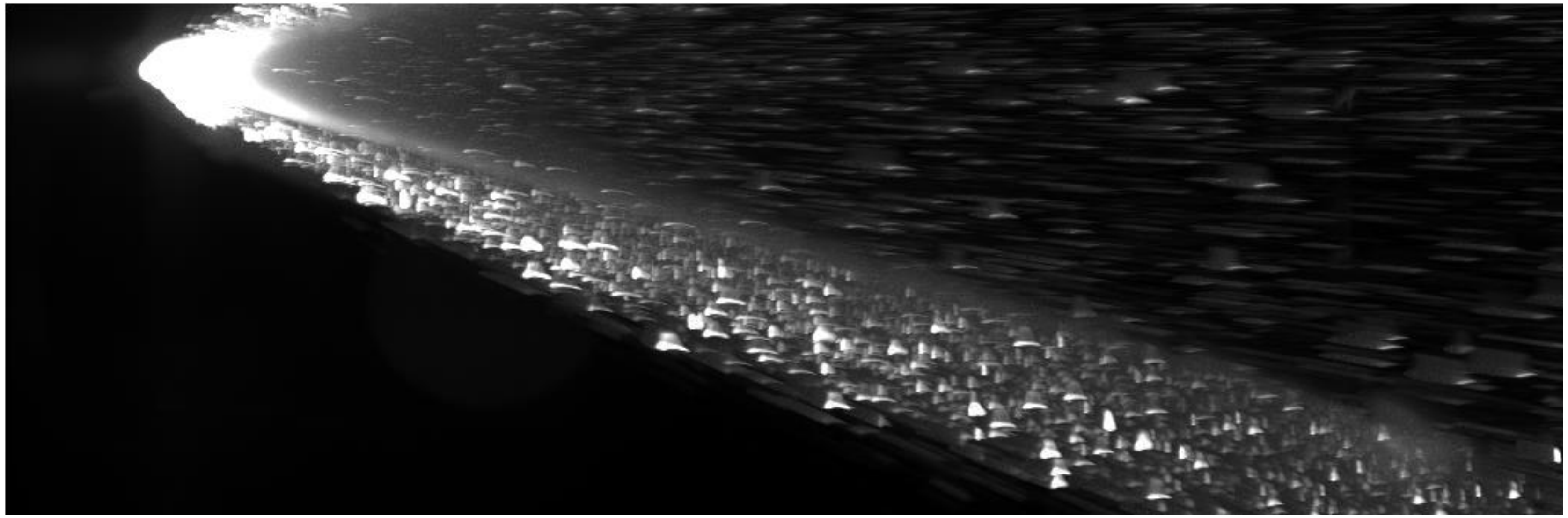
AHF Test Campaign / Spallation Test

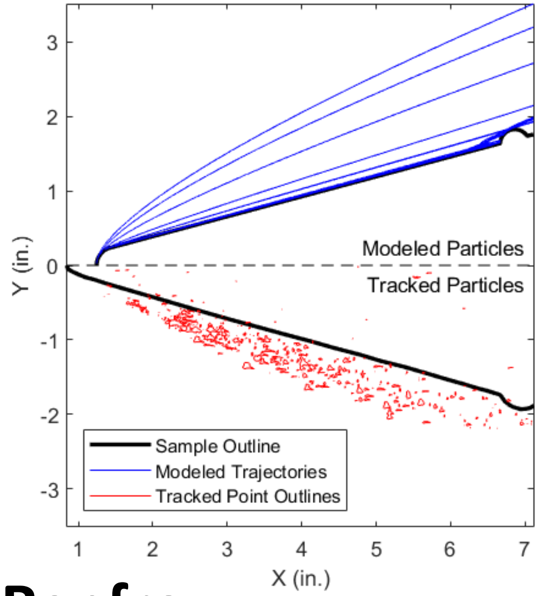
- **Spallation testing:**
  - **Compiled high-speed images match with pre-test spallation models**
  - **Physically captured particles are within bounds of previously estimated particle sizes**











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PICA-NuSil

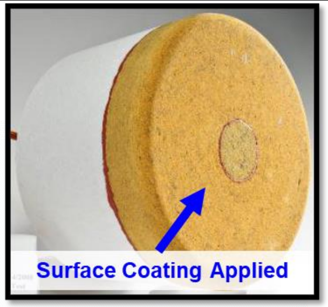
MSL Heat Shield

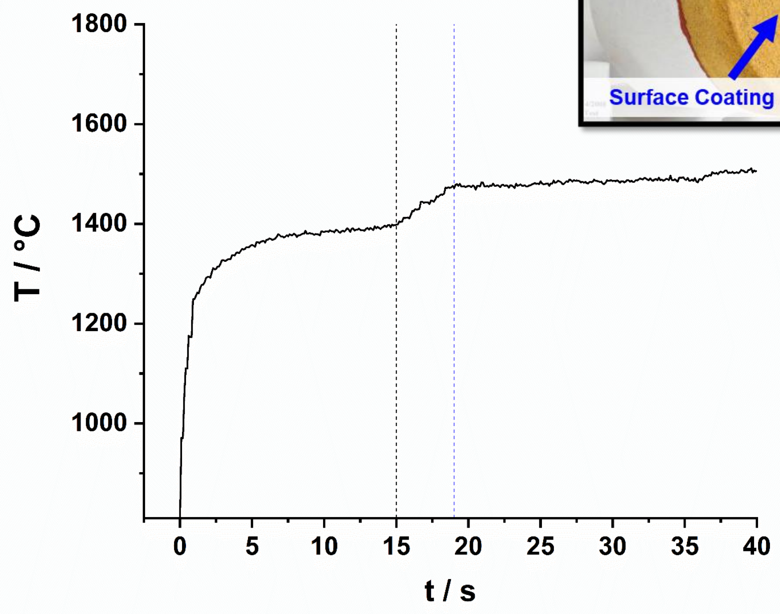
- **PICA is friable.**

NuSil (CV-1144-0)

- **An overcoat of NuSil (CV-1144-0) is applied to the surface to mitigate phenolic particulate shedding.**

Surface-Temperature Discontinuity



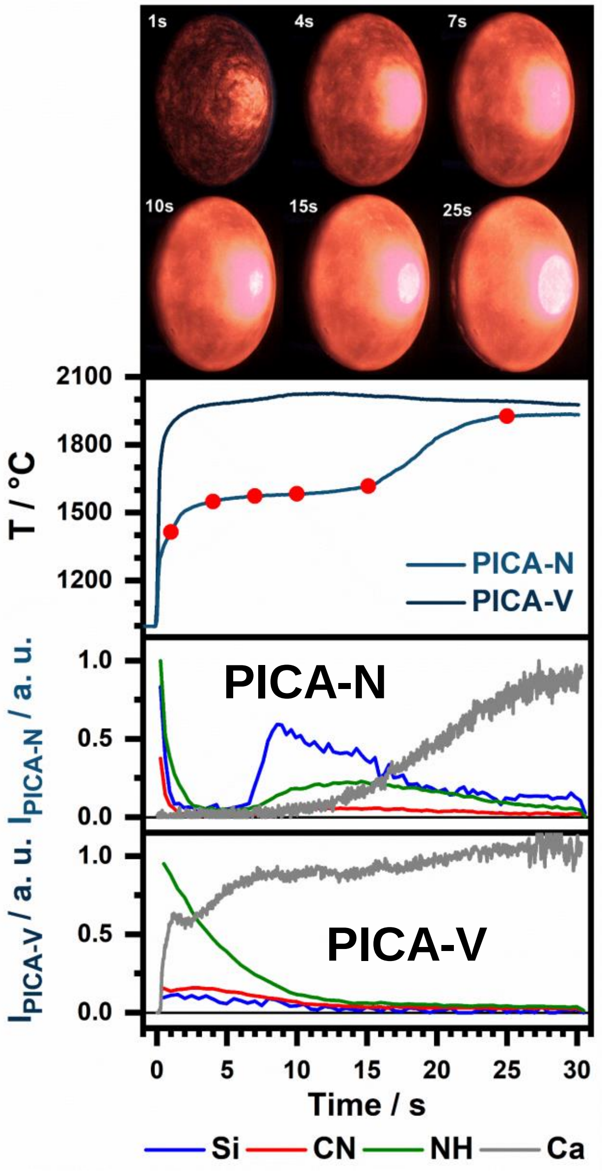


Surface Temperature of PICA with , NuSil Coating. (85 W cm^{-2})

- **State of the art material response models are under development to account for the surface temperature phenomenon.**

- **Jeremie Meurisse, “3D ablation modeling of silicone-coated heatshield compared to MEDLI2 in-flight data” – tomorrow at 2:00 P.M. (TP-18)**

PICA-NuSil



- Mini sphere-cone models of PICA-NuSil were subjected to arc-jet testing at the HyMETS facility located at NASA Langley in Virginia.

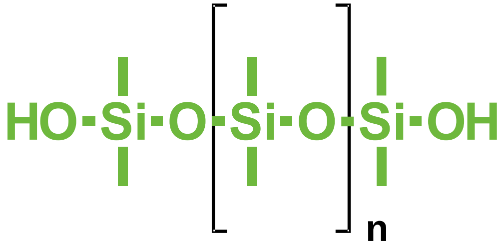
- Pyrometer data, emission spectra, and high-speed video reveal that the coating survives on the surface for several seconds before decomposing.

- A second round of HyMETS experiments will focus on testing PICA-NuSil in the CO_2 environment.

PICA + NuSil

+

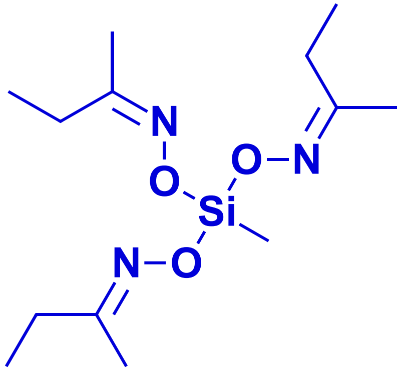
Siloxane Copolymer Backbone



n ≈ 262 ± 6 monomer units

+

Oxime Crosslinking Agent

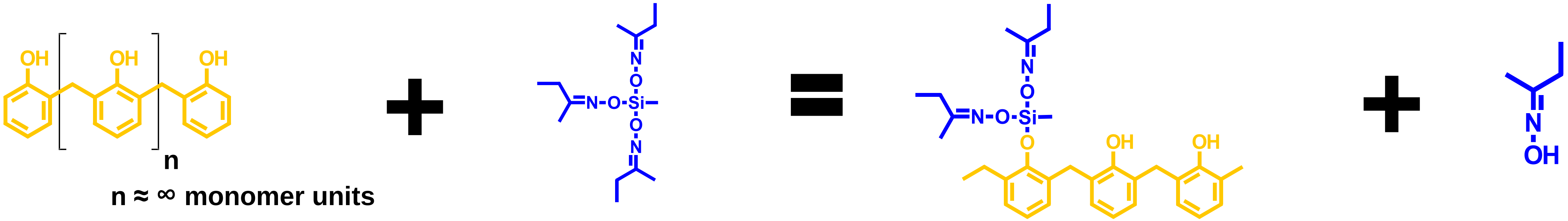


= ?

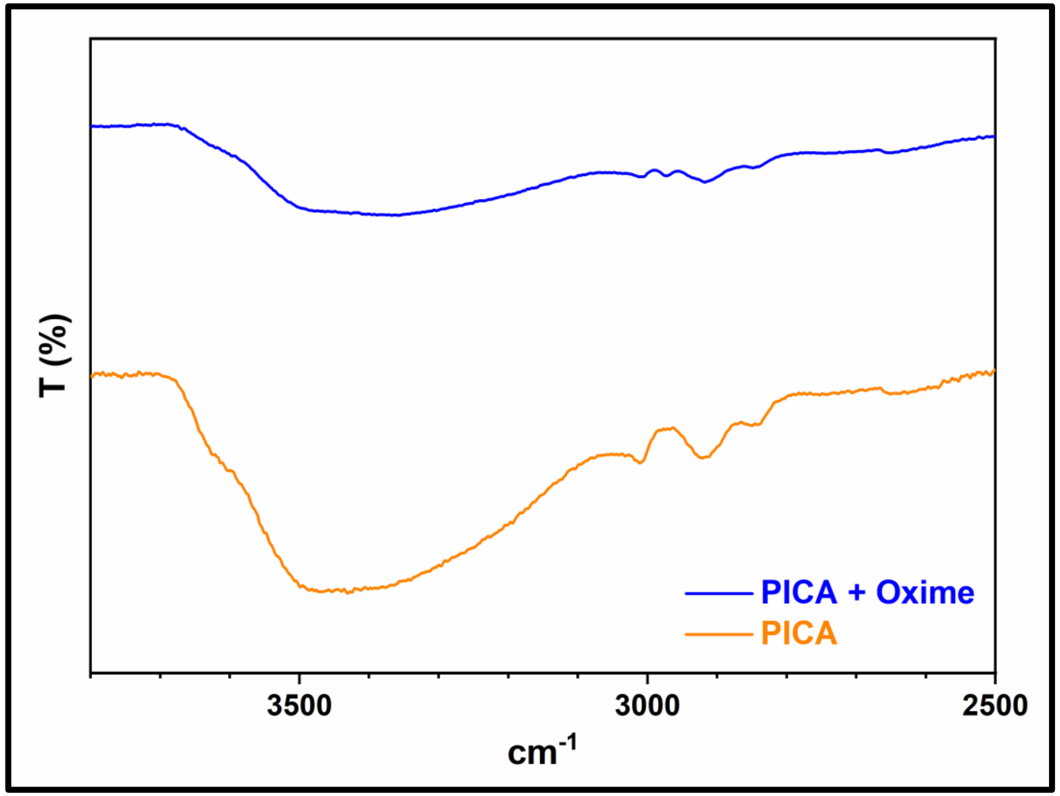
Do components of NuSil react with PICA?

Possible Reactions Under Ambient Conditions

Crosslinking compounds reacts with hydroxyl functional units of phenolic resin.



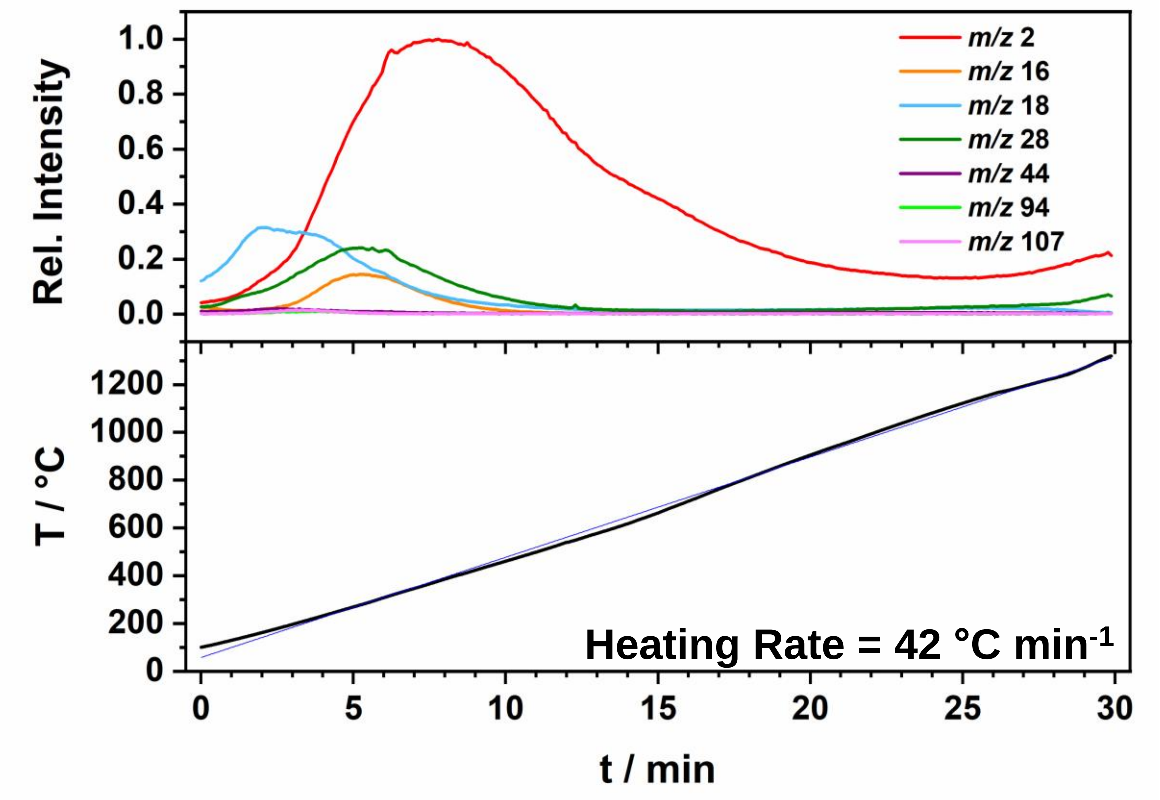
- **5 wt. % oxime mixed with naphtha.**
- **2 mm. dia. rod of PICA soaked in solution overnight.**
- **PICA + oxime cured in open air for 1 week.**
- **Attenuated Total Reflectance (ATR) spectra reveal a decreased intensity of OH stretch (3000 cm^{-1} – 3550 cm^{-1}).**



PICA

- **Dominant pyrolysis products:**
  - **H_2, CH_4, H_2O, CO.**
  - **Signal from polymer backbone fragments (e.g., phenol, cresol).**
  - **m/z 18 – Water evolves as a product of ether bond formation between hydroxyl functional groups of the phenolic polymer.**
  - **m/z 16, 28 – Peak evolution of methane and carbon monoxide at <u>t ≈ 5.5 minutes</u>.**
  - **m/z 2 – Peak evolution of Hydrogen at 7.7 min.**
  - **m/z 2, 28 – Hydrogen and Carbon Monoxide rise again after T > 1,100 °C.**

<u>Thermal Decomposition of PICA</u>



- **Char yield typically 80 wt. %.**

Pyrolysis Gas Testing

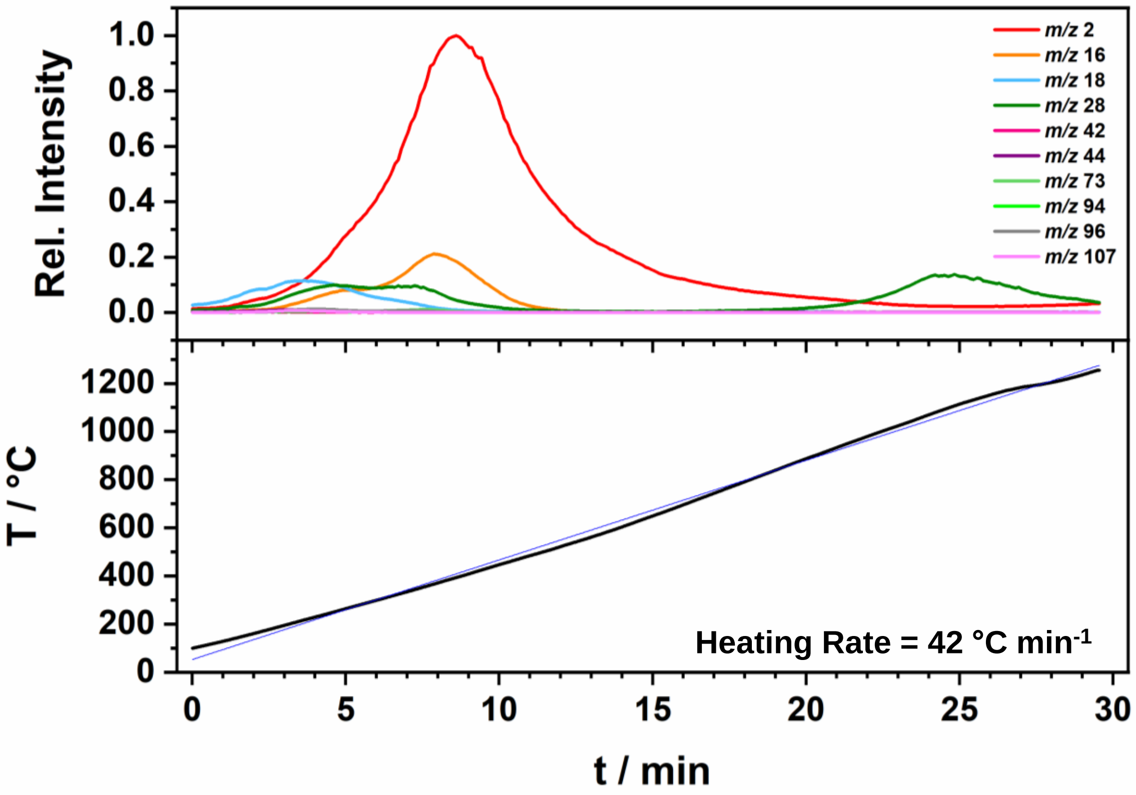
PICA + 45 wt. % NuSil

- Mixed 45 wt. % of NuSil with naphtha.
- 2 mm dia. rod of PICA soaked in NuSil solution overnight.
- PICA + NuSil solution cured in open air for 1 week.

Dominant pyrolysis products

- H_2, CH_4, H_2O, CO.
- *m/z* 18 – Low temperature evolution of water is bimodal. Evolution of water may be reduced in the presence of NuSil.
- *m/z* 2, 16 – Evolution of hydrogen and methane are bimodal. Peak methane production is shifted to higher temperature.
- *m/z* 28 – Evolution of carbon monoxide at low and high temperature.

Thermal Decomposition of PICA-N



- NuSil reacts with PICA at high temperature.

End